Chemistry Unit
Procedures for the Use of Reference Materials and Known Materials

1 Purpose

The Chemistry Unit (CU) uses reference materials for qualitative and quantitative analysis. This document describes the CU's procedures for the acquisition, storage, and verification of reference materials. Refer to the *CU Procedures for Measurement Traceability* for information regarding the establishment of measurement traceability through the use of reference materials.

A known material is an item acquired for the purpose of comparison with an evidentiary sample (e.g., commercial products, items received directly from manufacturers). This differs from a reference material in that only the source of the known material, not the exact composition, needs to be known at the time of acquisition. This document describes the information that will be recorded when using a known material in casework.

2 Scope

This document applies to CU personnel that acquire, store, and/or verify reference and/or known materials for use in casework.

3 Equipment/Materials/Reagents

The appropriate equipment, materials, and reagents used to verify a reference material will depend upon the nature of the substance. Since most of these verifications will be performed following an established CU standard operating procedure (SOP), the equipment, materials, and reagents required for such will be listed within the SOP used.

Known materials will undergo the same relevant analytical examinations that are performed on a questioned sample(s) during casework. The equipment, materials, reagents, and other relevant information may be found in the applicable SOP(s) being used.

4 Procedures

4.1 Purchasing a Reference Material

A *Requisition for Supplies and/or Equipment* (FD-369 or equivalent) will be prepared for all reference materials to be purchased. All FD-369 forms (or equivalent) for reference materials will be approved by the Unit Chief prior to ordering. CU's Chemical Inventory Manager (CIM)

will be notified of reference material purchases/receipts through the CU Chemical Products database, or by receiving a copy of the FD-369 (or equivalent) from the CU purchase credit card holder.

When appropriate, a certified reference material will be purchased. When a reference material is received in the CU, the CIM will verify that a Certificate of Analysis (COA) is requested/received from the manufacturer, if available.

4.2 Storage of Reference Materials

The reference material will be stored following manufacturer's recommendations.

4.2.1 Storage of Controlled Substances

If the reference material received is a controlled substance, the initial product weight will be recorded electronically in the CU Chemical Products database.

All controlled substances (with the exception of low concentration solutions, such as 1 mg/mL reference material solutions) will be stored in Redacted which is an evidence storage room (ESR) secured for dual-person entry. When Redacted to acquire a reference material, the *Access Log – Evidence Storage Facility* (FD-455) will be filled out.

4.3 Opening Reference Materials

4.3.1 Opening Controlled Substance Reference Materials

When opening a controlled substance reference material for the first time, it is good laboratory practice to record the date and your initials directly on the container; however, this is not a required practice. Each time any amount of a controlled substance reference material is removed from its container, the before and after weights of the container will be recorded in the CU Chemical Products database.

4.3.2 Opening Non-Controlled Substance Reference Materials

When opening non-controlled substance reference materials for the first time, it is good laboratory practice to record the date and your initials directly on the container; however, this is not a required practice.

4.4 Synthesis of a Reference Material

When a suitable reference material is not available from a vendor, it may be necessary to synthesize it. The following information will be recorded and provided to the CIM.

- The procedure used to synthesize the material

- The date of synthesis
- The initials of the person who synthesized it
- Any unique storage requirements
- Controlled substance schedule, if applicable

4.5 Reference Material Verification

Certified reference materials do not require verification. Additionally, Metallurgy maintains a reference collection that does not require verification. These reference materials are accompanied by certificates that justify the scope of their use, however the certificates do not meet the requirements to allow the materials to be classified as certified reference materials.

For all other non-certified reference materials, only one sample per manufacturer's lot number must be verified. Subsequent reference materials from the same lot will be considered as having the same verification as the original. The identity of the reference material will be verified prior to, or in concurrence with casework as described below.

4.5.1 Identity Verification

Use one or more of the following techniques to verify the identity of the reference material:

- Gas Chromatography/Mass Spectrometry (GC/MS)
- Liquid Chromatography/Mass Spectrometry (LC/MS)
- Direct Analysis in Real Time/Time-of-Flight Mass Spectrometry (DART/TOFMS)
- Gas Chromatography (GC) with applicable detector(s)
- Pyrolysis Gas Chromatography/Mass Spectrometry (Py-GC/MS)
- Fourier Transform Infrared Spectroscopy (FTIR)
- Raman Spectroscopy
- Ultraviolet-Visible Spectroscopy (UV-Vis)
- Powder X-ray Diffractometry (XRD)
- Scanning Electron Microscopy with Energy Dispersive X-ray Spectrometry (SEM/EDS)
- X-ray Fluorescence Spectroscopy (XRF)
- Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)
- Inductively Coupled Plasma/Optical Emission Spectroscopy (ICP/OES)
- Ion Chromatography (IC)

Prior to use, verify that the above instrument(s) is in proper working order by following the instrument's *Performance Monitoring Protocol*. Instrumentation not listed above may be used, provided it is shown to be in proper working order prior to use. When the identity verification is completed, provide the applicable data and instrumental parameters to the CIM.

4.5.2 Discrepancies in Identity

Discrepancies in the structural identity of a reference material following qualitative testing will be discussed with the supplier and the material returned, if applicable. If the material is identified as something other than intended, the CIM must be notified. If the material is retained, the container must be labeled with information indicating the discrepancy. The supporting data will be provided to the CIM.

4.6 COAs and Other Records

COAs and other records associated with reference materials may be obtained through multiple sources. Records may be received in physical form along with the reference material. Alternatively, the records may be downloaded in electronic format from the supplier or manufacturer website. The applicable records and verification data will be maintained in the CU.

4.7 Records for Known Materials

When known materials are used in casework, sufficient information will be recorded in the case notes such that the nature of the known material is established. Examples of the type of information that may be necessary to record, if applicable, include:

- Product name
- Manufacturer name
- Universal Product Code (UPC)
- Lot number
- Expiration date
- Location purchased/acquired
- CU unique identifier for database samples

5 Calculations

Not applicable.

6 Measurement Uncertainty

The *CU Procedures for Estimating Measurement Uncertainty* provides guidance for accounting for the uncertainty associated with the purity or concentration of reference materials.

7 Limitations

The limitations associated with this procedure are dependent on the instrumental techniques used to determine the identity and purity of reference materials. In general, the listed techniques will be sufficient for these determinations when performed as described.

8 Safety

Take precautions for the handling of all chemicals. Refer to appropriate SDS for safe handling practices. Refer to the *FBI Laboratory Safety Manual* for guidance.

9 References

CU Procedures for Measurement Traceability

FBI Laboratory Operations Manual

CU Procedures for Estimating Measurement Uncertainty

FBI Laboratory Safety Manual

Clarke's Analysis of Drugs and Poisons, Pharmaceutical Press (multiple editions, also available online)

The Merck Index, RSC Publishing (multiple editions, also available online)

Instrumental Data for Drug Analysis, T. Mills, J.C. Roberson, C.C. Matchett, M.J. Simon, M.D. Burns, and R.J. Ollis, Jr., 3rd ed., Volumes 1-6, CRC Press: Boca Raton, Florida, 2006.

Rev. #	Issue Date	History
8	09/13/19	Removed "Subunit" from "Metallurgy" in first paragraph, and edited the details of Metallurgy reference materials for clarity. Changed "shall" to "will" in section 3 (second paragraph, first sentence). Revised section 4.1 to include the CU database and purchase credit card holder responsibility. Revised second paragraph of section 4.1 for clarity. Removed the use of the CU Controlled Substance Log from section 4.2.1 and remaining sections including previous Appendix A (weight information will be recorded in CU database). Removed several storage requirements from section 4.2.1 since the requirements are no longer necessary. Added last bullet to section 4.4. Removed the need to print applicable information to be provided to the CIM in section 4.5.1. Changed "MSDS" to "SDS" in section 8.
9	07/15/20	Removed verification content from section 1 and merged with similar content in section 4.5 to remove redundancy. Revised scope to include Fire Debris. Minor edits to section 3, defined SOP acronym. Edited section 4.5.1 for clarity and updated instrument list, used instrument acronyms throughout rest of document. Removed previous section 4.5.2 (Purity Verification) as CRMs will be used for quantitations and do not require verification. Likewise, removed previous section 4.6.2 (Discrepancies in Purity) and content in section 5 (Calculations). Renamed section 4.5.2 and deleted previous section 4.6 for consolidation purposes. Deleted last sentence from section 4.6.1. Added "or concentration" to section 6.

Approval

Redacted - Signatures on File

Fire Debris Technical Leader: Date: 07/14/2020

General Chemistry Technical Leader: Date: 07/14/2020

Metallurgy Technical Leader: Date: 07/14/2020

Paints and Polymers Technical Leader: Date: 07/14/2020

Toxicology Technical Leader: Date: 07/14/2020

Chemistry Unit Chief: Date: 07/14/2020

QA Approval

Quality Manager: Date: 07/14/2020